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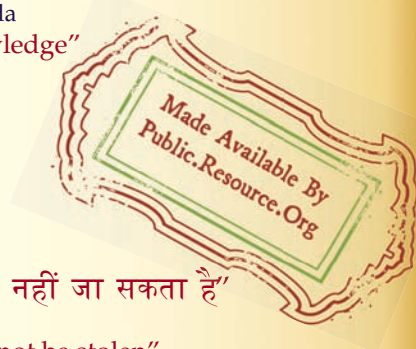
IS 4336 (1985): 4-Chloroaniline [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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(Reaffirmed 2000)

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Indian Standard
SPECIFICATION FOR 4-CHLOROANILINE
(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 5

Indian Standard

SPECIFICATION FOR 4-CHLOROANILINE

(First Revision)

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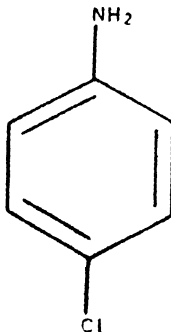
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Indian Standard
SPECIFICATION FOR 4-CHLOROANILINE
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 January 1985, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 4-Chloroaniline (C_6H_5NCl) is used in the production of intermediates and dyestuffs. It has the following structural formula:



4-CHLOROANILINE
(Molecular Mass 127.57)
(CAS No. 106-47-8).

0.3 This standard was first published in 1967. The committee responsible for the preparation of this standard decided to revise it in order to stipulate the requirement of maximum limits of impurities such as aniline, 2-Chloroaniline, 3-Chloroaniline and others. Thin layer chromatographic test method has been incorporated in this version for estimation of impurities.

0.4 This edition 2.1 incorporates Amendment No. 1 (May 2002). Side bar indicates modification of the text as the result of incorporation of the amendment.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in

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accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 4-Chloroaniline.

2. REQUIREMENTS

2.1 **Description** — The material shall be in the form of a yellowish to brownish crystals.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4-CHLOROANILINE

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO
(1)	(2)	(3)	(4)
i)	Crystallizing point °C, <i>Min</i>	69.5	A-1
ii)	Moisture content, percent by mass, <i>Max</i>	0.5	A-2
iii)	Matter insoluble in dilute, hydrochloric acid, percent by mass, <i>Max</i>	0.2	when tested as per IS : 5299 2001
iv)	Purity by nitrite value, percent by mass, <i>Min</i>	99	A-4
v)	Impurities, percent by mass, <i>Max</i> :		
	a) Aniline	0.5	A-5
	b) 2-chloroaniline	0.5	
	c) 3-chloroaniline	0.5	

3. PACKING AND MARKING

3.1 **Packing** — Unless otherwise agreed to between the purchaser and the supplier the material shall be packed in suitable steel drums (see IS : 2552-1979†).

3.2 **Marking** — Each container shall be securely closed and shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his trade-mark, if any;
- Lot or batch number;
- Gross, net and tare mass; and
- Pictorial marking as per IS : 1260 (Part 1)-1973‡.

*Rules for rounding off numerical values (revised).

†Specification for steel drums (galvanized and ungalvanized) (second revision).

‡Pictorial markings for handling and labelling of goods: Part I Dangerous goods (first revision).

3.2.1 Each container shall in addition bear the minimum cautionary notice worded as under:

‘AVOID CONTACT WITH SKIN, DO NOT HEAT
TO DECOMPOSITION’

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE -- The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — Representative samples of the material shall be drawn as prescribed in Appendix B.

5. TEST METHODS

5.1 Test shall be conducted according to the methods prescribed in Appendix A. References to relevant clauses of Appendix A are given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

A P P E N D I X A

(*Table 1, Clause 5.1*)

METHODS OF TEST FOR 4-CHLOROANILINE

A-1. DETERMINATION OF CRYSTALLIZING POINT

A-1.1 Apparatus

A-1.1.1 The apparatus of the shape, dimensions and tolerances given in Fig. 1 consists of the following.

*Water for general laboratory use (*second revision*).

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A-1.1.2 Outer Glass Test Tube --- serves as an air jacket and is weighed with lead shots or a similar loading material. It is provided with a cork through which the inner tube (*see* A-1.2) is held in position.

A-1.1.3 Inner Glass Test Tube — fitted with a cork which carries a stirrer in the form of a loop of glass with a glass stem and the thermometer placed centrally within the tube and the glass loop. The bottom of the bulb of the thermometer shall be about 10 mm from the bottom of the inner glass test tube. The cork is so fixed that the immersion mark on the thermometer is level with the top of the cork.

A-1.1.4 Cooling Bath — 1 000-ml beaker about 150 mm in height. The level of the cooling liquid in the bath shall be at least as high as the level of the sample in the inner tube.

A-1.1.5 Thermometer --- conforming to IS : 2480 (Part 1)-1983*.

A-1.1.5.1 Any other thermometer of similar range and accuracy may also be used. The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

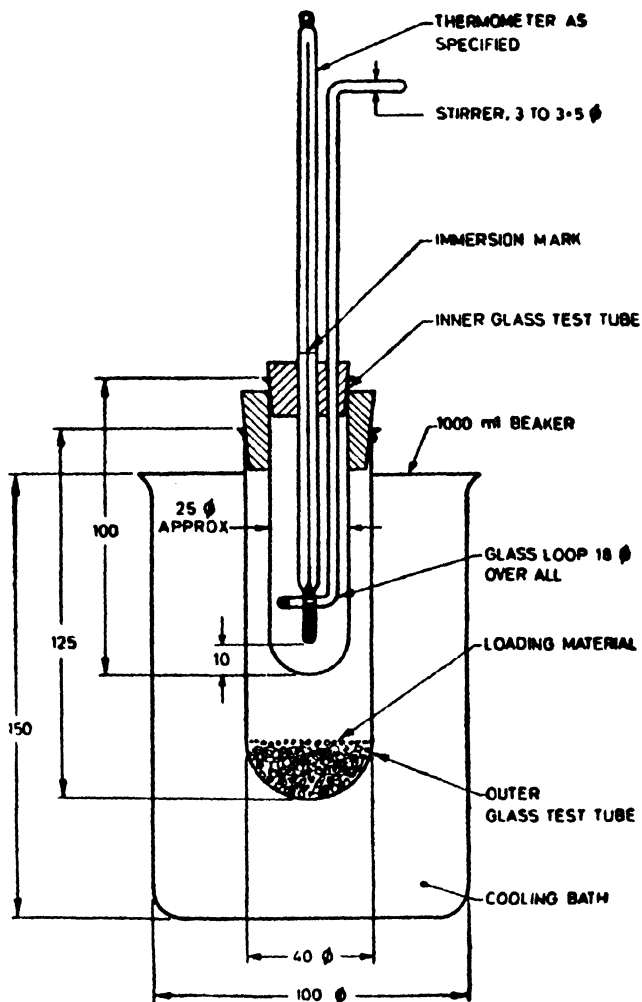
A-1.2 Procedure

A-1.2.1 Weigh as quickly as possible about 40 g of the material in a conical flask. Loosely stopper the flask and warm it in a water-bath at about 85°C. Allow the crystals to melt completely. Mix the molten material thoroughly and transfer a quantity (about 20 g) of the material into the inner tube of the crystallizing point apparatus sufficient to cover the bulb of the thermometer. Add a few crystals of the material for seeding. Replace the inner tube in its jacket and assemble the apparatus as shown in Fig. 1, with the cooling bath between 6 to 8°C below the expected crystallizing point.

A-1.2.2 Note the thermometer readings at intervals of half a minute, with continuous and gentle stirring. The crystallizing point corresponds to the first five consecutive readings during which the temperature remains constant within 0.1°C.

A-1.2.3 If the supercooling takes place as shown by the rise in temperature, the constant temperature shall be observed immediately after the temperature rise. A temperature rise of 1°C shall be regarded as the maximum allowable.

*General purpose glass thermometer: Part 1 Solid stem (*second revision*).



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF CRYSTALLIZING POINT

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A-1.2.4 If a constant temperature is not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature rise. The point of intersection shall in this case be taken as the crystallizing point.

A-2. DETERMINATION OF MOISTURE CONTENT (DEAN AND STARK METHOD)

A-2.1 Apparatus

A-2.1.1 *Flask* — 500 ml capacity, of the shape as shown in Fig. 2, made of hard resistance glass, well annealed and as free as possible from striae and similar defects.

A-2.1.2 *Condenser* — made of hard resistance glass, well annealed and as free as possible from striae and similar defects, with the shape, dimensions and tolerances as shown in Fig. 3.

A-2.1.3 *Two-millilitre Receiver* — made of hard resistance glass, well annealed and as free as possible from striae and similar defects, provided with ground glass joints, with the shape, dimensions and tolerances given in Fig. 4, consisting essentially of the upper chamber together with the tube and ground joint leading to the flask, and the graduated tube.

A-2.1.3.1 The graduated portion shall have a capacity of 2 ml at 20°C when filled to the highest graduation mark. The scale shall cover the range of 0.1 to 2 ml and shall be divided into intervals of 0.05 ml. The graduation marks corresponding to 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml shall be numbered. The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.15 ml, 0.25 ml, 0.35 ml and so on up to and including 1.95 ml, shall be carried half way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each and beyond the shortest graduation marks. The error at any point on the scale shall not exceed ± 0.03 ml and the difference between the errors at any two points shall not exceed 0.03 ml.

A-2.1.3.2 When a metal flask is used, care has to be taken to provide an airtight connection between the flask and the receiver.

A-2.1.4 *Graduated Cylinder* — 100 ml capacity.

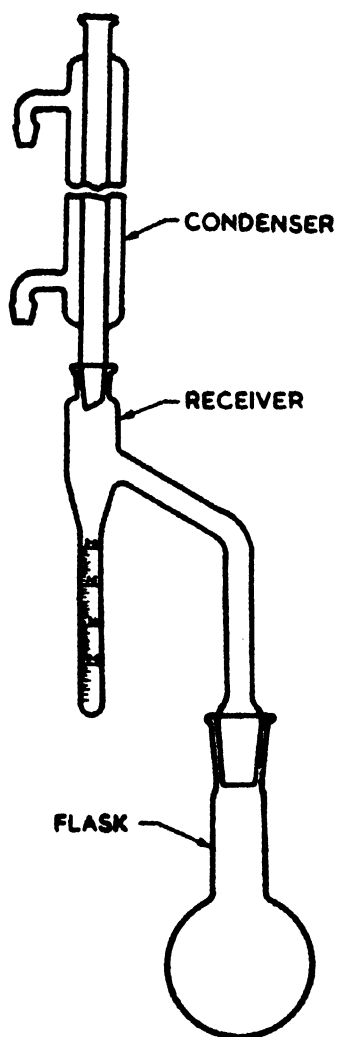
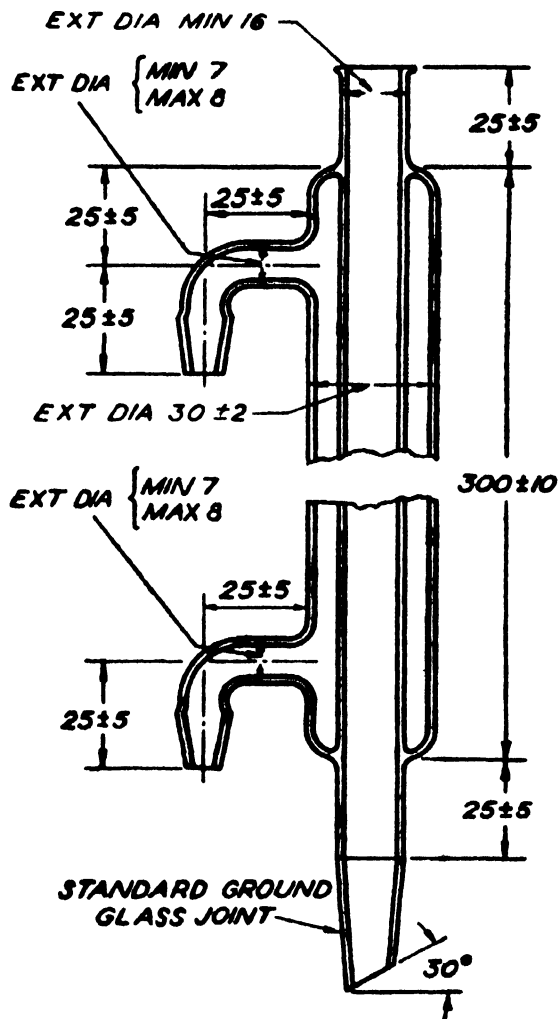


FIG. 2 DEAN AND STARK ASSEMBLY



All dimensions in millimeters.

FIG. 3 CONDENSER

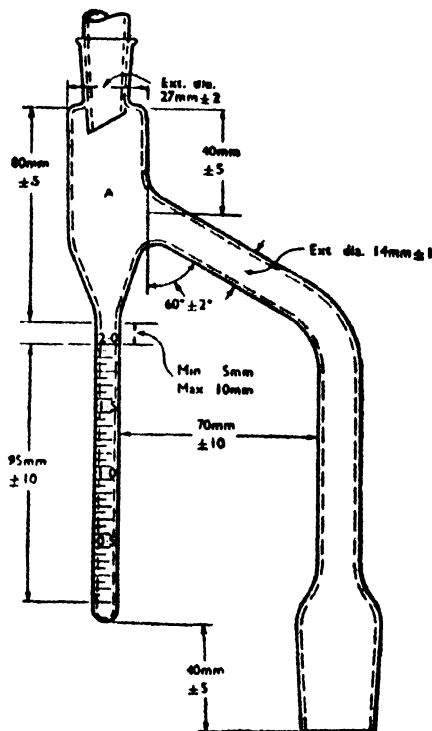


FIG. 4 TWO-MILLILITRE RECEIVER

A-2.2 Procedure — Weigh, as quickly as possible, 100 g of the powdered material directly into the flask and add 100 ml of solvent naphtha light (conforming to IS : 213-1968*). Attach the flask to the Dean and Stark condensing and collecting system and heat the flask at such a rate that condensate falls from the end of the condenser at a rate of 2 to 5 drops per second. Continue the distillation until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. Remove a persistent ring of condensed water in the condenser tube, if any, by increasing the rate of distillation by a few drops per second. Wash droplets of water which adhere to the lower end of the condenser tube into the receiver with solvent naphtha, light, using any suitable means.

*Coal based naphtha (*first revision*).

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A-2.2.1 Note the number of millilitre of water in the receiver at the room temperature. Assuming that one millilitre of water is equal to one gram, report as percentage as by mass in the material.

A-3. SOLUBILITY IN HYDROCHLORIC ACID

A-3.1 Weigh accurately about 2 g of the material and add 100 ml of 10 percent (v/v) hydrochloric acid. Stir to dissolve completely, warm if necessary. Not more than a slight opalescence shall be observed.

A-4. DETERMINATION OF PURITY

A-4.1 Outline of the Method — 4-Chloroaniline is estimated by direct titration with standard sodium nitrite solution using starch iodide paper as indicator.

A-4.2 Reagents

A-4.2.1 Sodium Nitrite Solution — approximately 0.2 N, freshly standardized.

A-4.2.2 Starch Iodide Papers

A-4.2.3 Concentrated Hydrochloric Acid — conforming to IS : 265-1976*.

A-4.3 Procedure — Accurately weigh by difference from a weighing bottle about 10 g of the material and transfer to a beaker containing 400 ml of water at about 60°C and 50 ml of hydrochloric acid, stir to dissolve completely, warming further, if necessary. Cool to 20°C. Make up to 1 000 ml in a volumetric flask.

Transfer by means of pipette 100 ml of the above solution into a beaker containing 200 g of crushed ice and 200 ml water with 5 g of potassium bromide and 20 ml concentrated hydrochloric acid. While stirring mechanically titrate with sodium nitrite solution as rapidly as possible to a distinct blue ring test on a starch iodide paper. The end point is taken when the test persists for 5 minutes without further addition of nitrite.

A-4.4 Calculation

$$\text{Purity, percent by mass} = \frac{127.57 \times V \times N}{M}$$

where

V = volume in ml of sodium nitrite solution used,

N = normality of sodium nitrite solution, and

M = mass in g of material used in the test.

*Specification for hydrochloric acid (*second revision*).

A-5. DETERMINATION OF IMPURITIES

A-5.0 General — The impurities in 4-Chloroaniline is determined by ascending thin layer chromatographic method.

A-5.1 Apparatus

A-5.1.1 Micropipette

A-5.1.2 Developing Chamber

A-5.1.3 Thin Layer Chromatographic Glass Plates — 20 × 20 cm.

A-5.1.4 Chromatographic Sprayer

A-5.1.5 Adjustable Thin Plate Applicator

A-5.1.6 Weighing Bottle

A-5.1.7 Oven

A-5.2 Reagents

A-5.2.1 Silica Gel G — Neutral

A-5.2.2 Developing Solvent — Benzene : Chloroform (95 : 5) (v/v).

A-5.2.3 Sample Solution — 1 percent solution in methanol.

A-5.2.4 Reference Solution

- a) 0.005 percent solution of 100 percent aniline in methanol,
- b) 0.005 percent solution of 100 percent 2-chloroaniline in methanol, and
- c) 0.005 percent solution of 100 percent 3-chloroaniline in methanol.

A-5.2.5 Spray Reagent — N-1-Naphthylethylenediamine dihydrochloride (1 percent in water).

A-5.3 Procedure

A-5.3.1 Preparation of Chromatographic Plates — Mix about 50 g of neutral silica gel G with 85 to 90 ml of water for three minutes to form a uniform slurry. Then by means of the applicator, coat the plate with this slurry to get silica gel G layer of thickness not exceeding 0.25 mm. Activate it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel G from either of the lateral sides of each plate.

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A-5.3.2 Chromatographic Separation — Pipette 10 μ l of the sample solution (see A-5.2.3) and 10 μ l of reference solutions (see A-5.2.4) and apply in the form of a uniform spot on the plate at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the developing solvent (see A-5.2.2) to run up to 14 cm at room temperature. Take out the plate and dry completely. Keep the plate for 2 minutes in a chamber containing nitrous acid fumes (prepared by mixing sodium nitrite and hydrochloric acid). Blow out the excess nitrous fumes from the plate and spray with the spraying reagent (A-5.2.5). The material and the constituent impurities are identified by their respective characteristic colours as under:

<i>Substance</i>	<i>Zone</i>	<i>RF Value</i>	<i>Colour</i>
Aniline	I	0.31	Bluish-Violet
4-Chloroaniline	II	0.38	Blue
3-Chloroaniline	III	0.47	Red
2-Chloroaniline	IV	0.67	Brownish-red

A-5.4 Reporting — Report impurity as that which is nearest in intensity to the reference samples. In ease, the colour intensity does not come in the range of the standard spots of impurity, repeat the whole procedure using suitable concentrations of various impurities.

APPENDIX B

(Clause 4.1)

SAMPLING OF 4-CHLOROANILINE

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument, and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B.2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided consisting of two concentric cylindrical tubes made of a metal and which is not affected by the material being sampled (preferably of stainless steel) closely fitting each other throughout their entire length so that one tube can be rotated within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position, the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sample is usually provided with locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most needs.

B-2.1.1 Use of Sampling Instrument — The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case of minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same container such that they are at least 75 mm in the case of kegs, drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

B-3. SCALE OF SAMPLING

B-3.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, then the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED
FOR SAMPLING

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 15	2
16 .. 25	3
26 .. 50	4
51 and above	5

B-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case, such tables are not available, the following procedure may be adopted.

Starting from any container, count them in one order as 1, 2, 3.....up to r and so on, where r is the intergral part of N/n (N being the lot size and n being the number of containers to be selected). Every r th container thus counted shall be withdrawn to give sample for test.

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From each of the containers selected as in B-3.2, draw with an appropriate sampling implement small portions of the material from different parts of the container. The total quantity so drawn from each of the container shall be approximately equal to thrice the quantity required for testing purposes as indicated in B-4.2.

B-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

B-4.3 From the samples (*see B-4.2*) representing different containers selected in **B-3.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out testing for the characteristics specified under **B-6.2**. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-4.4 The remaining portion of the material in the samples (*see B-4.2*) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-4.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **B-1.7**.

B-4.6 The referee sample consisting of a composite sample and a set of n individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Tests for the determination of moisture content and purity shall be conducted on each of the individual samples (*see B-4.4*).

B-5.2 Tests for the other requirements given in **2.1** and Table 1 shall be conducted on the composite sample (*see B-4.3*).

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of purity and moisture content, if each of the individual test results satisfy the relevant requirements given in Table 1.

B-6.2 Composite Samples — For deciding the conformity of a lot to the requirements of all other characteristics tested on the composite sample (*see B-4.3*), the test results for each of the characteristics shall satisfy the relevant requirements given under Table 1.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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